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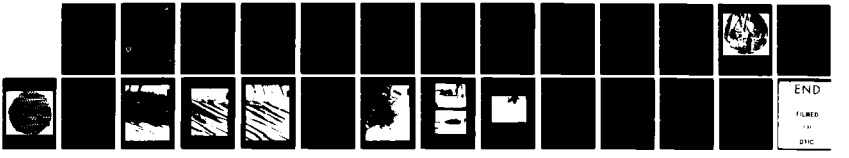

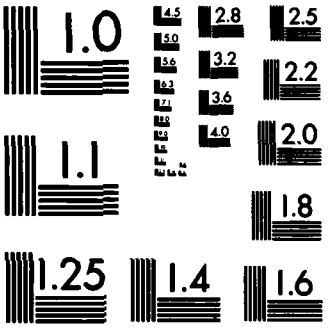

MEMORANDUM REPORT ARBRL-MR-03217

EXAMINATION OF OPTICAL HOLOGRAMS BY A SCANNING ELECTRON MICROSCOPE

Charles R. Stumpfel

November 1982

US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
BALLISTIC RESEARCH LABORATORY
ABERDEEN PROVING GROUND, MARYLAND

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER Memorandum Report ARBRL-MR-03217	2. GOVT ACCESSION NO. AD-A122 335	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Examination of Optical Holograms by a Scanning Electron Microscope		5. TYPE OF REPORT & PERIOD COVERED 6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Charles R. Stumpfel		8. CONTRACT OR GRANT NUMBER(s) -
9. PERFORMING ORGANIZATION NAME AND ADDRESS US Army Ballistic Research Laboratory ATTN: DRDAR-BLT Aberdeen Proving Ground MD 21005		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 1L161101A91A
11. CONTROLLING OFFICE NAME AND ADDRESS US Army Armament Research & Development Command US Army Ballistic Research Laboratory (DRDAR-BL) Aberdeen Proving Ground, MD 21005		12. REPORT DATE November 1982 13. NUMBER OF PAGES 26
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

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17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Holography
Scanning Electron Microscope Examination
Silver Halide Emulsions

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Two optical holograms recorded in silver halide photographic emulsions were examined by a scanning electron microscope. Surface relief produced by the light exposure as well as in-depth silver fringes (also lightwise) were imaged. Other structure, some of which is periodic - but none of which is known to be produced by the light - is pointed out and possible causes of the patterns mentioned.

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I. INTRODUCTION

When objects which are located in scattering aerosols such as smoke are photographed or holographed the photons which contain the object information are recorded simultaneously with a high background of scattered photons. Noise in the recording and readout of photographic recordings limits the depth into the scattering aerosol to which photographed objects may be detected.

It seems possible that a scanning electron microscope if used to read out information recorded in a photographic emulsion (or near its surface) might yield useful object information not available with the ordinary method of read out-by photon illumination of the hologram or photograph. In addition, the technique of combining optical holography and scanning electron microscope examination appears to be a convenient potential method for producing (by holography) and examining (by SEM) the smallest possible light patterns. This capability may be useful in the future for fundamental studies of the relationship of photons, the recording media and interference.

Previous publications of scanning electron microscope examinations of holograms (other than holographically produced gratings) were not found so our first step was to examine the simplest holographic exposures. Photographs or holograms of objects recorded simultaneously with scattered light backgrounds have not yet been examined.

Photographic emulsions consist of silver halide crystals in gelatin. Exposure to light forms "specks" or clusters of a few atoms of silver in the crystals. In the development process the silver halide crystals with one or more specks are catalyzed to atomic silver at higher rates than the silver halide crystals without specks. The silver halide crystals which are not transformed are removed by fixing. After exposure and processing the finished emulsion is composed of gelatin and grains of atomic silver. In addition, emulsion surface relief resulting from spatial variations of the light intensity is also observed.[1]

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[1] *H. M. Smith, JOSA, V 58, N4, April '68.*

Common photographic emulsions, containing silver halide crystals about 1 micrometer in size, have resolutions on the order of 100 cycles/millimeter. Holographic emulsions, with crystal dimensions ranging from 0.03 to 0.08 micrometers, have much higher resolutions. The resolution is usually stated to be "greater than 2,000 cycles/millimeter." Meaningful "figures of merit" with specific measuring techniques for the resolutions of holographic emulsions have not found common usage. One estimate of resolution, "6000 cycles/millimeter" (based on recording Argon laser fringes), is offered for comparison to the resolutions of the coarser emulsions.[2] In any case, the silver halide grains of holographic emulsions can record light patterns which vary over dimensions considerably smaller than a wavelength of light.

The objective here was to produce small structure in holographic exposures and examine it with a scanning electron microscope (SEM). Thereby structure, particularly any recorded near the intersection of the emulsion surface and photon field, might be observable with sub-wavelength resolution of, say, 0.1 micrometer or better.

A model Super III A scanning electron microscope, manufactured by International Scientific Instruments, Inc., was used to study two holograms. The accelerating voltage was always 30 Kv. This instrument uses a very small diameter ($\sim$0.01 micrometer) beam of monoenergetic electrons to bombard the surface of a specimen. This causes other electrons to be emitted from the specimen. When the emitted electrons are classified by their energy, two main components may be identified: 1) a secondary electron component [defined as electrons with less than 50 ev. energy], the energy distribution of which peaks below 5 ev.; and 2) a backscattered electron component the energy distribution of which peaks at the energy of the scanning electrons (30 Kev). The signal used to produce our images, except Figure 4 - Left Side, originates mostly from the secondary electrons. The emitted electrons are detected and the resulting signal is used to vary the beam intensity of a cathode ray tube which is x-y coordinated with the scanning electron beam.

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[2] *Kodak Pamphlet No. P-110*

The secondary electron image of the surface is often considered a "topographical" rendition of the specimen. However, to be topographical the secondary electron "escape depth"[+] must be much smaller than the dimensions of the topographical features. Estimates of the "escape depth" (for the simple case of an E-beam normally incident on a featureless surface) for secondary electrons range from 0.01 to 0.05 micrometers and for the backscattered electrons range from 1 to 2 micrometers.[3,4] To further complicate interpretation of the "image", identical surfaces (atomic composition, orientation, etc.) may yield different signals wherever the beam strikes near adjacent structure at angles which cause the beam to travel just under a nearby surface allowing much shorter electron escape paths (brightens that location of image). For example, for beam entry points near ridge peaks secondary electrons may be detected by escaping out the side from as far as a few micrometers below the beam entry point. If, on the other hand, the beam strikes in a valley the secondaries may be captured by the specimen before detection (darkens that location of image).

Understanding of the interaction of an electron microscope and these silver grain/gelatin emulsions is not very advanced and therefore we have not attempted to measure the dimensions of image features.

Some of the more important factors which will contribute to secondary electron micrographs of silver halide holograms are listed below:

1. Hologram surface relief is known to be produced by recording fringes. (The D-19 used for developing is a non-tanning developer [tanning, a cross linking of gelatin molecules, is known to increase surface relief]).[1]

2. Surface relief, independent of light exposure, occurs on the emulsions from manufacturing, processing, handling, etc.

3. A high atomic number gold/palladium coating, which we found necessary to prevent specimen charging, was applied in a layer estimated to be less than 0.01 micrometers thick. Since this coating thickness is on the order of the "escape depth", a significant number of detected secondaries probably originate in the coating. The effect of the coating on our images is not very well understood.

4. The E-beam may change the specimen especially by evaporating the gelatin at higher rates than the silver.[5]

[+] *Escape Depth. For this discussion "escape depth" indicates that about 1-1/e or ~0.6 of the specified electrons (secondary or backscattered) originated within this depth of the surface.*

[3] *P.R.Thorton, The Scanning Electron Microscope, '68.*

[4] *O.C. Wells, Scanning Electron Microscope, '74*

[5] *S.J.W. Platzer, V.A.Greenhut, Photo.Sci. and Engin., V20,N6,Nov/Dec '76*

In addition, the SEM may be operated so that the image represents the backscattered electron signal by use of the "Robinson detection technique".++ Because silver atoms (atomic number = 47) have a higher backscattering coefficient[6] than the lower atomic number gelatin atoms (H,C,N,O;Z $\leq$ 8) the backscattered electron image enhances the location of silver grains. Also, because the escape depth is on the order of 1 micrometer, the backscattered image shows atom distributions from much farther below the surface. The only backscattered image is Figure 4 - Left Side.

II. HOLOGRAM PREPARATION

A. Hologram Photographic Processing

The following procedures were used in photographic processing of the holograms:

1. Hologram I (Plate, Kodak type 120-02, emulsion thickness ~6 micrometers)
    1. Develop: 6 minutes, D-19, 20°C
    2. Rinse: 30 seconds, Kodak Indicator Stop Bath
    3. Fix: 5 minutes, Kodak Rapid Fixer Solution A.
    4. Rinse: Several minutes in water
    5. Dry: Hang dry
2. Hologram II (Film, Kodak type SO-173, emulsion thickness ~6 micrometers)
    1. Develop: 6 minutes, D-19, 20° C
    2. Rinse: 30 Seconds, Kodak Indicator Stop Bath
    3. Fix: 5 minutes, Rapid Fixer
    4. Rinse: Water
    5. Rinse: 2 minutes,75% methanol/25% water (dye removal)
    6. Dry: Hang dry

B. Specimen Coating for SEM Examination

Hologram sections were removed from the plate by fracturing and from the film by cutting. To prevent E-beam charging the hologram sections were coated by sputtering with palladium/gold. The thickness of the coating is estimated to be less than 0.01 micrometer.

++*The "Robinson detection technique" involves placing a detector very close to the specimen to increase the solid angle of capture for the straight trajectory backscattered electrons.*

[6] *J. I. Goldstein, H. Yakowtiz, editors, Practical Scanning Electron Microscopy; Electron and Ion Microprobe Analysis, '75*

Repeated images of the same area of Hologram I, a plate, were identical indicating insignificant beam damage. An attempt to image Hologram II, a film, without a Pd/Au coating resulted in rapid E-beam damage. Viewed later by an optical microscope, fringe mounds, in the crossed pattern described later were seen to protrude from the eroded surface. See Figure 1. Hologram II was then Pd/Au coated and examined at high magnification. It is not known whether some damage continued to occur (and contributed to the electron micrograph to be shown in Figure 9) but the rapid evaporation experienced without coating was prevented. When examined by an optical microscope, the coating appears mostly intact, but some film distortion around the irradiated regions probably indicates the E-beam has induced stress in the polyester emulsion support (100 micrometers of Estar polyester).

Table 1 summarizes our preparation/damage experience. The glass mounted holograms appear to be resistant to damage at the E-beam current densities used.

TABLE I. PREPARATION AND TENDENCY TO DAMAGE

Hologram/Preparation	Result
Hologram I Plate, type 120-02 Pd/Au coated	Repeated scanning of the same area, at magnifications to 5,000, produced the same images.
Hologram II Film, Type SO-173 Uncoated	Rapid evaporation of gelatin leaving silver patterns (with one high current density scan).
Hologram II (Same specimen) Pd/Au coated	Damage reduced or eliminated. Probably limited to higher E-beam current densities.

III. DESCRIPTION OF HOLOGRAMS AND DISCUSSION OF ELECTRON MICROGRAPHS

A. Hologram I (Plate, Kodak type 120-02)

This type of hologram is termed an "image plane speckle reference beam hologram" and was produced with a two slit interferometer.[7] See Figure 2. The object was a back-illuminated ground glass against a rectangular aperture. Such an interferometer produces a speckled image of the ground glass with each speckle modulated by fringes with peak spacing of about 20 micrometers. See Figure 3, an optical micrograph. This is consistent with the formula for fringe spacing, δ_i:

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[7] *D.E.Duffy, Appl. Optics, V11, N8, Aug '72*

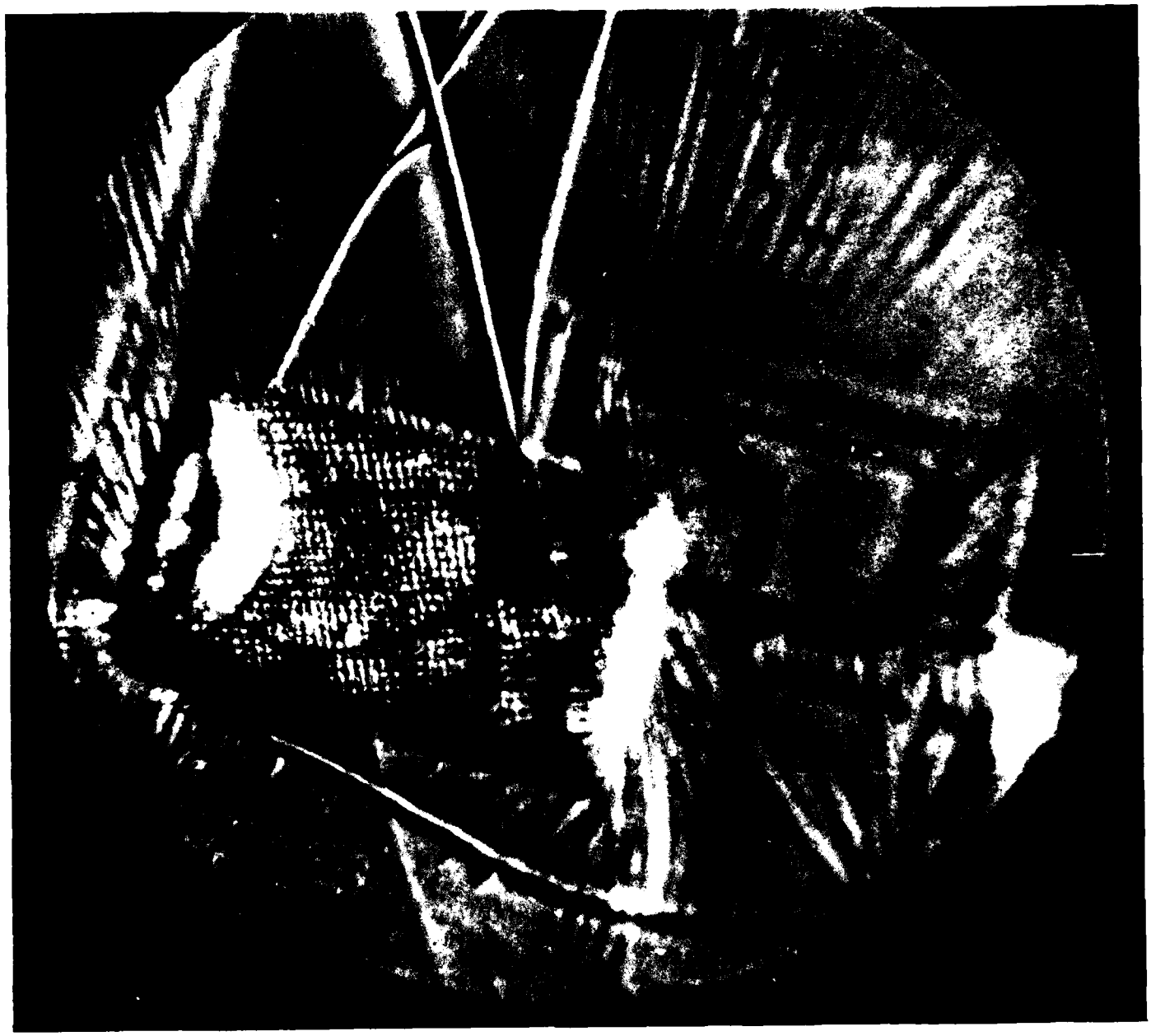

Figure 1. Optical Micrograph - Electron Beam Evaporation of Gelatin to Expose Mounds of Silver. Peak Spacing 4 Micrometers.

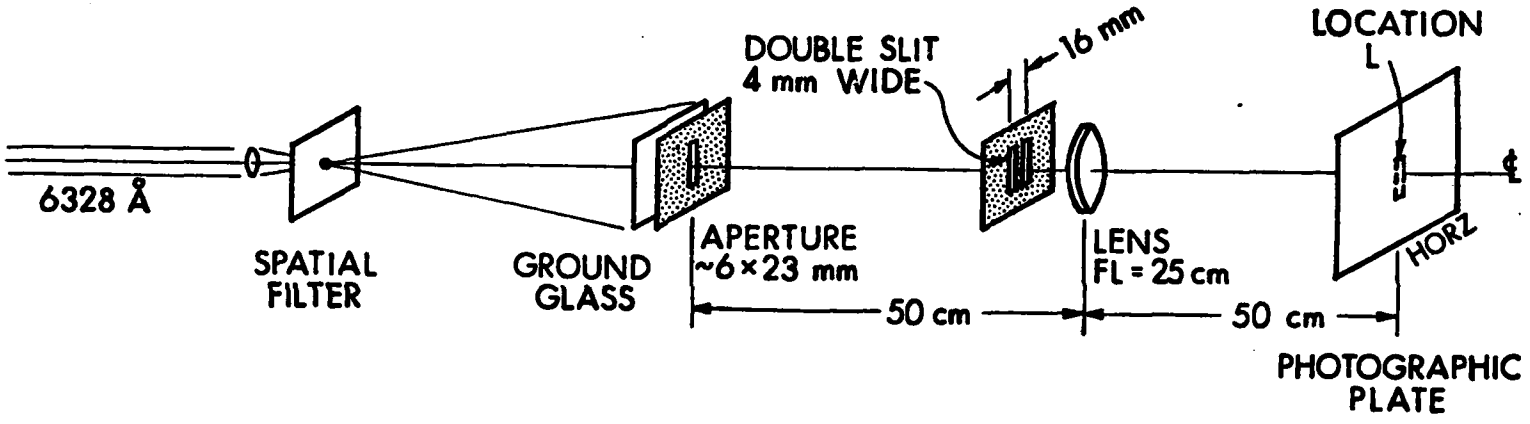

Figure 2 - Double Aperture Set-Up Used to Produce Hologram I.

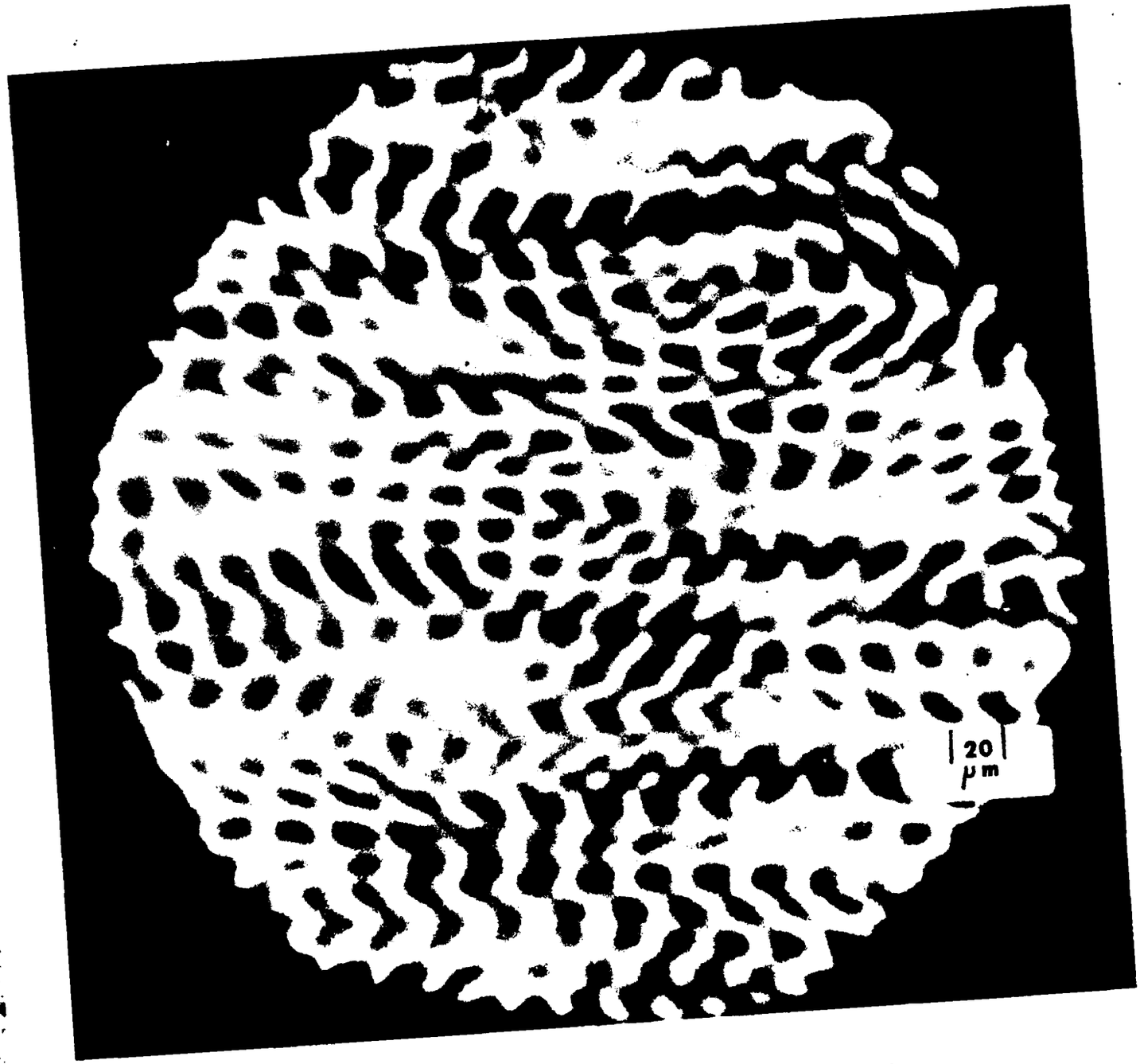

Figure 3 - Optical Micrograph of Hologram I

$$\delta_i = \frac{\lambda p m}{D}$$

$\delta_i \approx$ 20 micrometers

δ_i - peak-to-peak fringe spacing

p - object distance, 50 centimeters

m - magnification, 1

D - slit separation, 1.6 centimeters

λ - 0.6328 micrometer

Examined by eye, the exposed portion (image area corresponding to the ground glass) of Hologram I - after coating - is diffusely reflecting, indicating the emulsion has considerable surface relief. The light transmission of the hologram fragment examined by SEM was later estimated by measuring the light transmission of an adjacent uncoated piece of the same hologram. That piece transmits about 20% of incident white light (measured over a one millimeter area of emulsion).

An electron micrograph of Hologram I is shown in Figure 4 - Right Side. The best detail was obtained when the stage of the electron microscope was oriented to allow the E-beam to strike at a glancing angle to the surface. The 20 micrometer fringe peaks extend in a direction perpendicular to the broken edge of the emulsion. A set of corresponding peaks is indicated by *a*'s. Smaller periodic ridges, indicated by *b*'s, can be seen. The *b* structure is more apparent in Figure 5, a higher magnification image of the same area. For reference, a debris particle is identified by *p* in both Figure 4 and Figure 5. The *b* type structure is possibly standing waves in depth (Weiner structure) caused by reflections from the glass and viewed at a glancing angle. Or, since *b* structure is horizontally oriented, it might have been produced by spurious reflections from the optical table or another horizontal surface. These modulations occur in both peaks and valleys. A few smaller high contrast dark spots, indicated by *c*'s, are sometimes aligned with *b* structure minima. These spots could occur where the beam strikes in valleys as discussed. Likewise, the very high contrast ridge structure indicated by *d*'s which is aligned with the *b* structure may be *b* structure favorably oriented for capture of secondaries in the bottoms of the valleys. (The structure indicated by *m* is a scanning artifact of the electron microscope and can be seen to the right and left.)

The same region of the emulsion when viewed to enhance backscattered electrons (by detection with a "Robinson" detector) is shown in Figure 4 - Left Side. The same *a* peaks are marked. This image enhances the silver atoms and shows distributions from deeper below the surface. In the *original backscattered* E-micrograph, the *b* structure is faintly visible in a few locations; but it is indistinguishable in the report reproduction.

Figure 6 shows another pattern, two straight bars across the image indicated by *e*. These bars appear to intersect the *b* structure to produce smaller periodic structure. Again high contrast structure, similar to *d* type of Figure 5 is present and appears parallel to adjacent *b* structure.

Figure 4 - Electron Micrograph - Right - A Secondary Electron Image of Hologram I. Left - Corresponding Backscattered Electron Image.

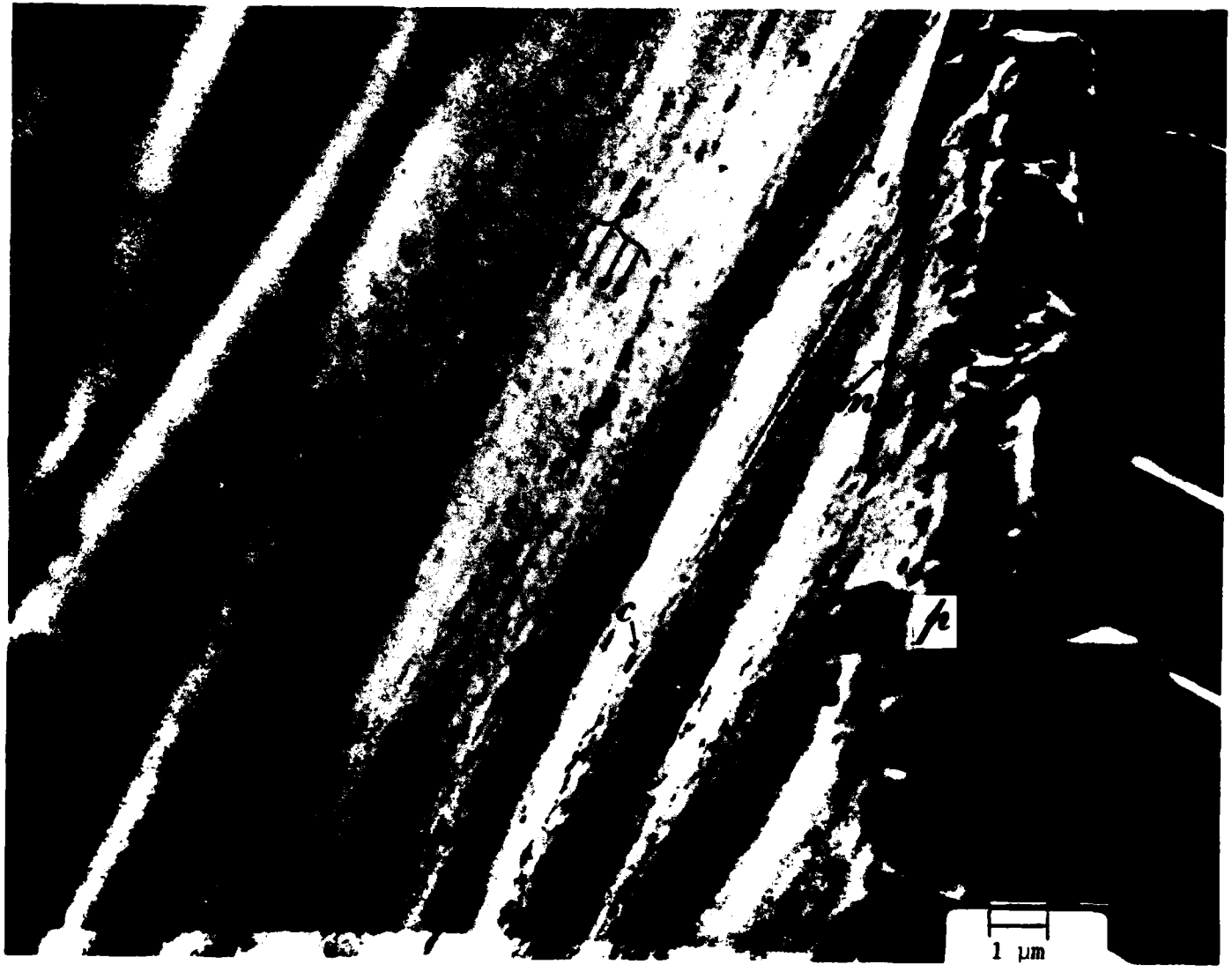

Figure 5 - Electron Micrograph - Higher Magnification Image of Hologram I Region Around Particle *p*.

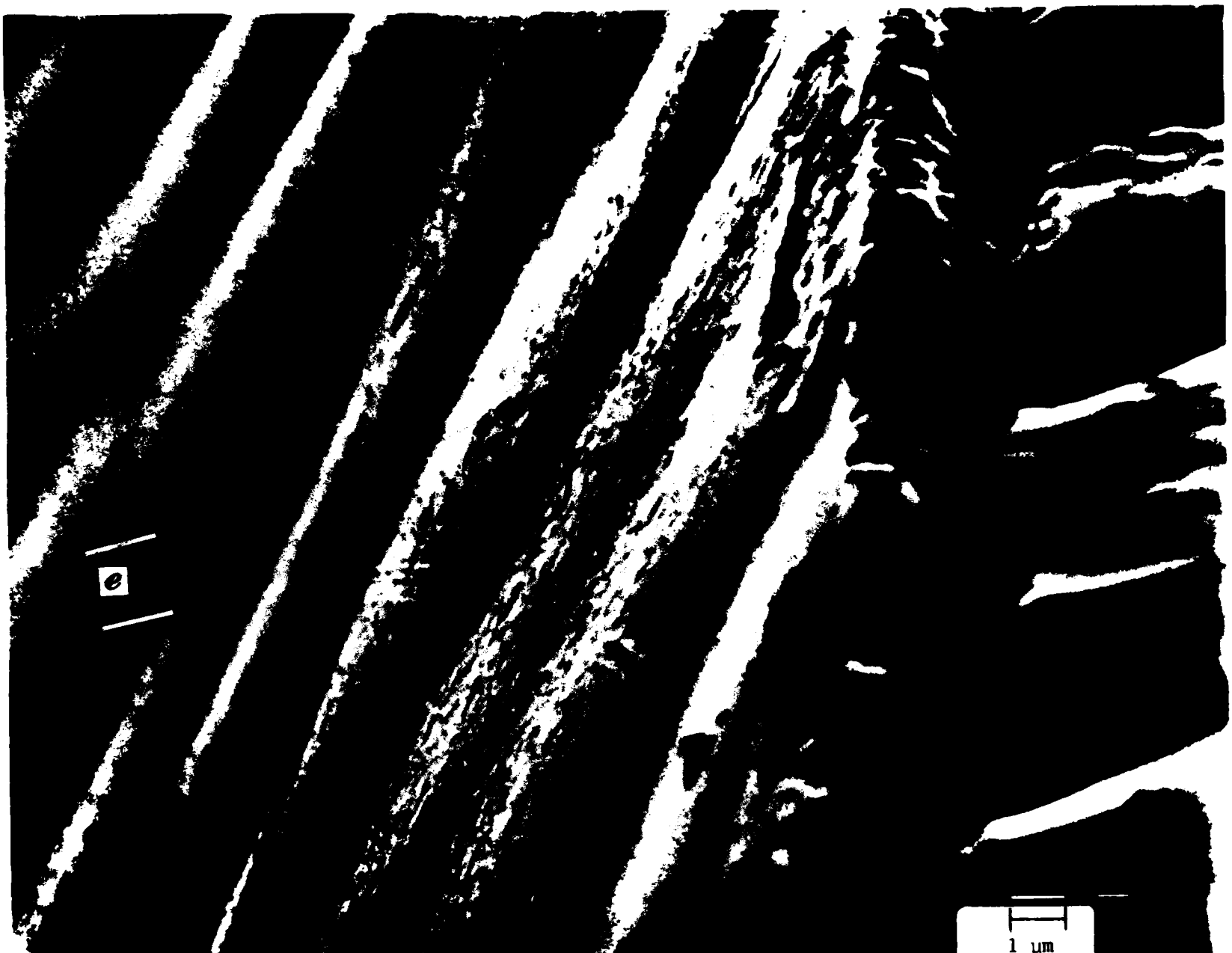

Figure 6 - Electron Micrograph - Another of Hologram I.

Figure 7 is an E-micrograph from the border region between the heavily exposed image of the ground glass (shown in Figure 4) and the little-exposed adjacent emulsion. This is the area of the image corresponding to the vicinity of a horizontal aperture edge (6 millimeter side). See location L of Figure 2. The structure - perhaps not lightwise - is not apparent at greater distances from the exposed region (This region may have received light from only one slit. These patterns have the horizontal orientation and roughly the shape one might expect from the intersection of a single slit/lens impulse response and the emulsion surface.)

B. Hologram II (Film, Kodak Type SO-173)

This is an off-axis hologram of a coin object with the object and reference irradiation impinging on the emulsion to produce fringes with average separation of about 4 micrometers. The taking set-up is shown in Figure 8. In order to have an easily identifiable pattern this hologram was double exposed with a 90^{o} rotation of the holographic plate between exposure (around an axis perpendicular to the film and passing through the center of the reference illumination). This produced two "crossed" fringe systems of high contrast when viewed on an optical microscope. The pattern is also displayed in the optical micrograph of gelatin evaporation damage, Figure 1.

When illuminated, both fringe systems holographically reconstruct the coin.

The E-micrograph of these patterns shows low contrast "crossed" fringes. See Figure 9.

IV. CONCLUSIONS

We have examined optical holograms recorded in silver halide emulsions with a scanning electron microscope. Identifiable patterns are easy to produce and examine. We have pointed out other structures, some periodic, which we have not yet identified or determined to be lightwise.

ACKNOWLEDGMENT

We would like to thank Gould Gibbons for applying his knowledge of the electron microscope to produce the micrographs.

Disclaimer: The identification of any product/manufacturer which appears in this report does not constitute an endorsement.

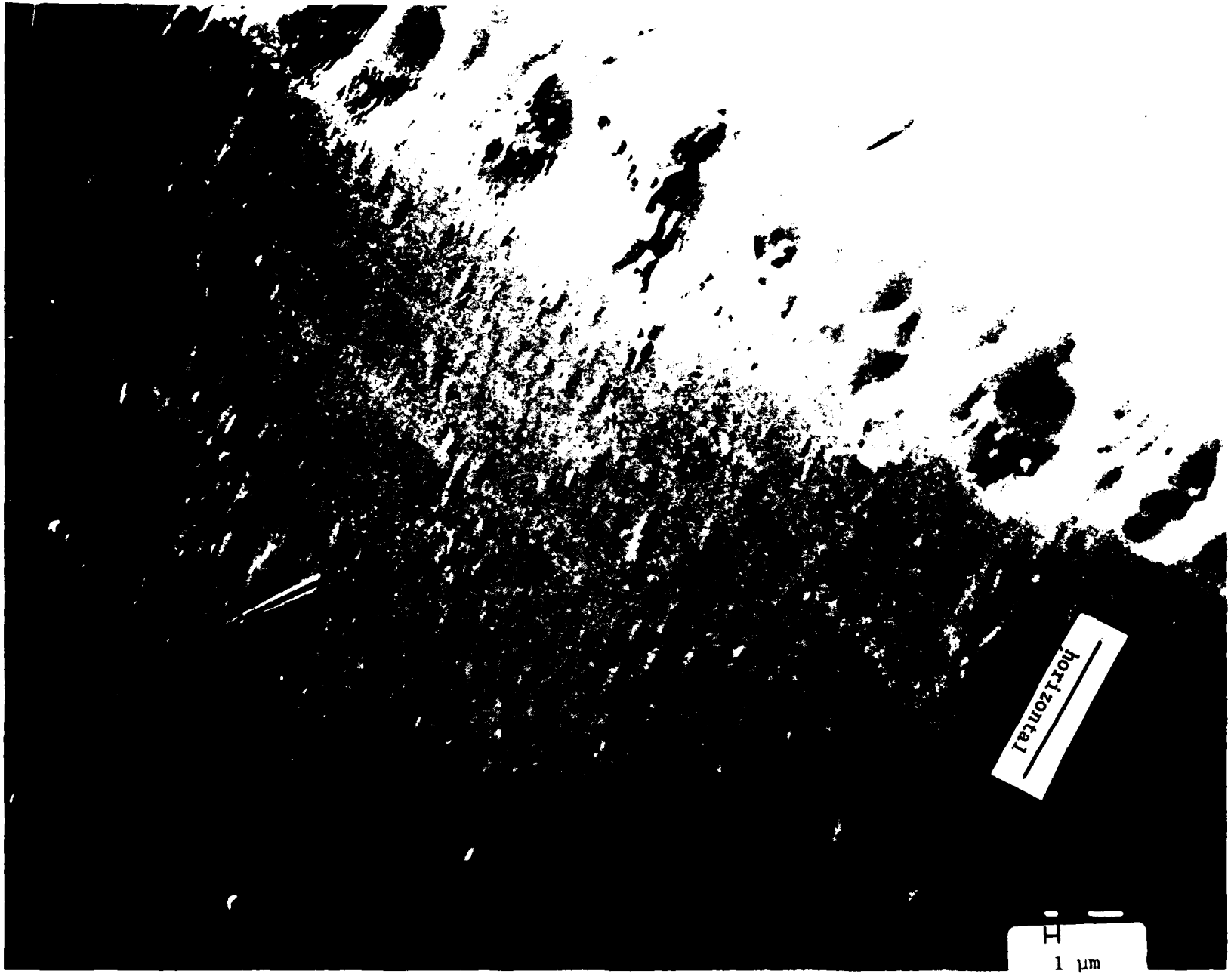

Figure 7 - Electron Micrograph - Vicinity Between Exposed and Unexposed Portions of Hologram I.

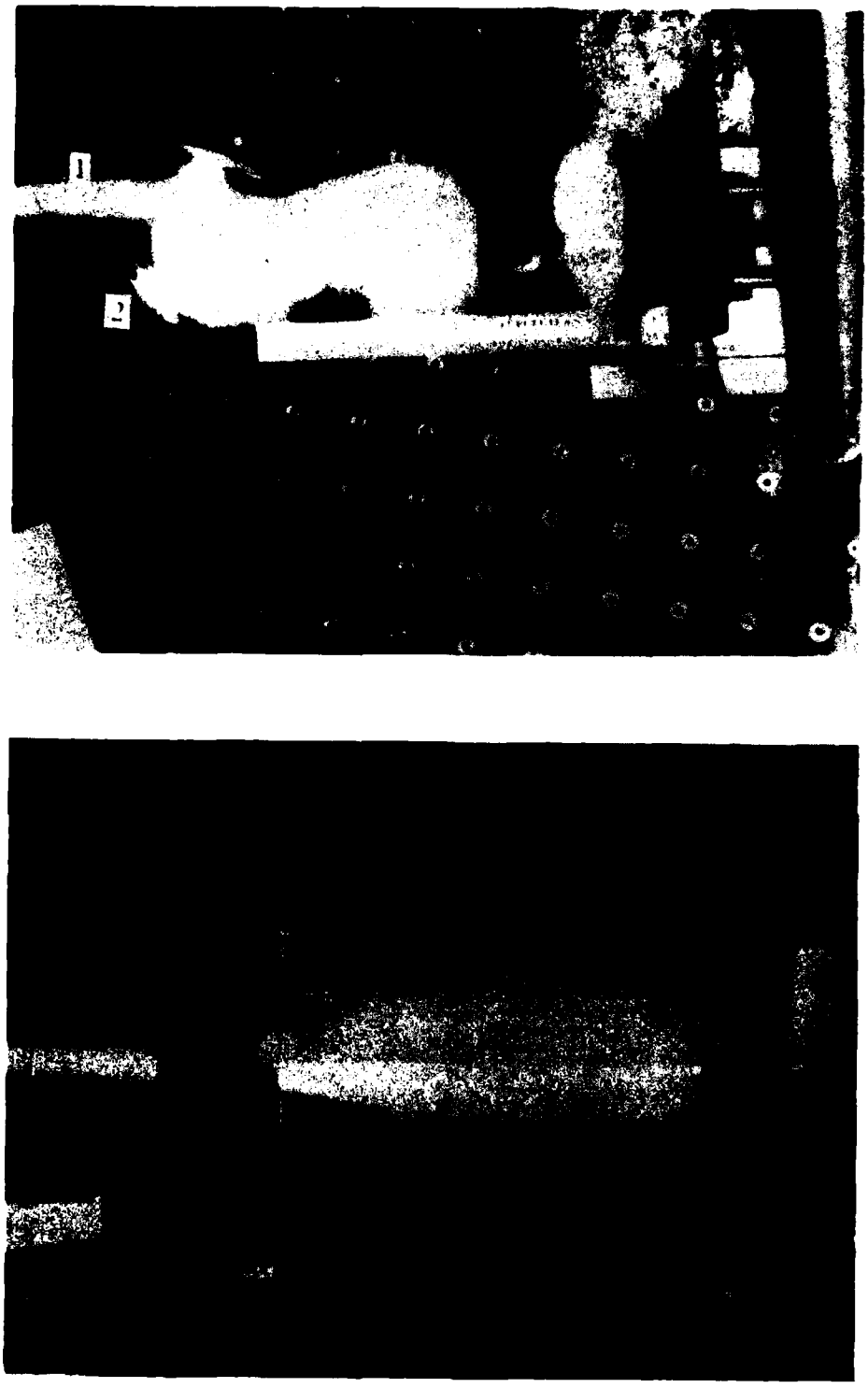

Figure 8 - Set-Up Used to Produce Hologram II. The Plate Was Rotated 90° Between Exposures.

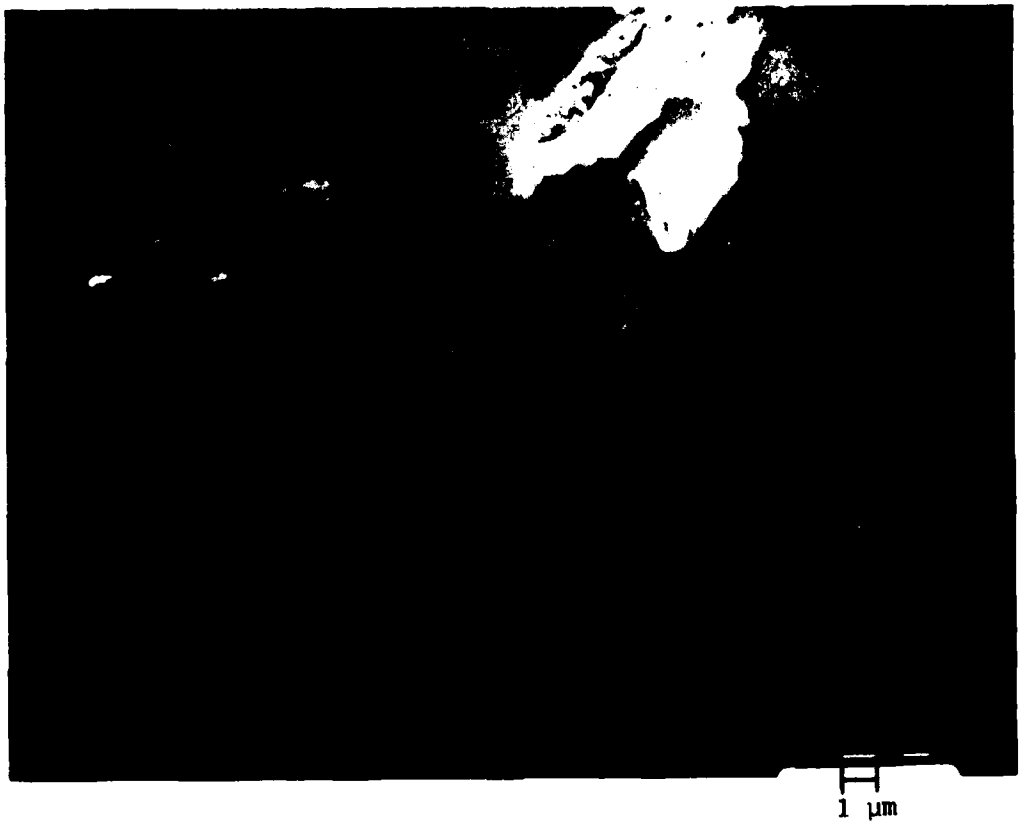

Figure 9 - Electron Micrograph of "Crossed" Fringe Pattern.

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